



## **Replacement Alternatives to the Chromate Wash Primer DOD-P-15328D**

**by Pauline Smith, Kestutis Chesonis, Christopher Miller,  
and John Escarsega**

**ARL-TR-3220**

**June 2004**

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Weapons and Materials Research Directorate, ARL**

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14. ABSTRACT The major advantage of the Department of Defense (DOD)-P-15328DD1 wash primer is to enhance corrosion resistance through the passivation of the metal surface. In the U.S. Army's Chemical Agent Resistant Coating (CARC) System, the wash primer DOD-P-15328D is overcoated with an epoxy primer and followed by a camouflage urethane topcoat. Several coating procedures specify the use of the wash primer, DOD-P-15328D, as a surface treatment prior to the application of an epoxy primer/polyurethane topcoat CARC system. The current wash primer contains large amounts of volatile organic compounds (VOCs) and hazardous air pollutants (HAPs) that impact coating operations due to air pollution regulations that may require the use of control devices to reduce the total VOC/HAP emissions to the atmosphere. The U.S. Army Research Laboratory (ARL) has evaluated new water-reducible wash primers that do not contain hexavalent chromium and significantly minimize VOC and HAP. Coatings have been extensively tested for accelerated corrosion and adhesion and have completed 2 years of outdoor exposure testing. ARL will conduct tests on military equipment to validate the lab and controlled testing previously completed. Affected installations, facilities, and weapons systems are excellent candidates for this evaluation because application of DOD-P-15328D is a key step prior to applying the remaining CARC system coating.					
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## 1. Introduction

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At the beginning of the last century, cleaned steel surfaces were passivated to corrosion by “washing” them with phosphoric acid solutions using sponges. In the 1930s, efforts to improve on the uneven application of the acid led to the use of polyvinyl butyral resins and chromate pigments for long-term corrosion inhibition. This technology became institutionalized during World War II when Union Carbide Corporation, under the direction of the U.S. Government, developed their wash primer, WP-1, (1) as a pretreatment for ship bottoms, with the patent assigned to the Secretary of the Navy. Their product evolved to the current specification, Department of Defense (DOD)-P-15328D (2). This specification has been used extensively by U.S. Armed Forces to treat ferrous and nonferrous surfaces that were not amenable to the pretreatment process, using immersion in multiple solution systems.

Specification DOD-P-15328D has a low-solids and high volatile organic compound (VOC) content, contains phosphoric acid with zinc chromate, and has hazardous air pollutants (HAPs). These characteristics come under the control of the National Ambient Air Quality Standards, Sections 109 and 112 of the Clean Air Act as amended in 1990. Over the years, the U.S. Army Research Laboratory (ARL) Coatings Technology Team has reformulated all of the camouflage chemical agent resistant coating (CARC) and ammunition coatings to meet local and Federal regulations. One of the most difficult tasks has been to reformulate a wash primer with reduced VOCs and zero HAPS that will have corrosion resistance similar to DOD-P-15328D without hexavalent chrome.

Wash primers are characteristically thin (0.3–0.5 mil [1 mil = 0.001 in]), cross-linked coatings applied directly to the substrate to provide protection from corrosion and promote adhesion (3). In the U.S. Army’s CARC System, the wash primer DOD-P-15328D is overcoated with an epoxy primer and a camouflage urethane topcoat. Several coating procedures specify the use of a wash primer, DOD-P-15328D, as a surface treatment prior to the application of an epoxy primer/polyurethane topcoat CARC system.

The CARC System application specification, MIL-C-53072C (4), requires that metal surfaces on tactical vehicles be treated to improve adhesion and corrosion resistance prior to coating with an epoxy primer and a camouflage topcoat. In original equipment manufacturer (OEM) processes, the surface treatment is generally performed by a five-stage dip process, e.g., zinc phosphate prescribed in TT-C-490 (5). In depot operations and for touch-up in OEM processes, the surface treatment requirement is met through the wash primer DOD-P-15328D. The formulation contains 7.1% zinc chromate and has 6.5 lb/gal of VOCs which are classified as HAPs. Based on the estimated usage of 21,000 gal/year, the following pollution is generated annually: 12,600 lb of zinc chromate and 35,700 gal of package/thinner solvents (6). This large amount of VOCs and HAPs directly impacts coating operations due to air pollution regulations that may

require the use of control devices to reduce the total emissions to the atmosphere. Because the pigmentation contains hexavalent chromium, the ARL Coatings Team has explored the use of alternative passivation. The reasons for hexavalent chromium substitutes are crucial—particularly for health, safety, environmental compliance, and pollution prevention.

Upon paint removal or stripping, the chromate wash primer, together with the CARC paint, generates hexavalent chromium-contaminated paint waste. This paint waste must be disposed as chromium-containing hazardous waste (7). Annually, ~6,000,000 lb of stripped CARC waste is produced. It costs ~\$0.61/lb to dispose of chromate-bearing paint waste, costing ~\$3,600,000 annually (5). It has been estimated that elimination of the chromate from the paint waste would eliminate the need to dispose of it as a hazardous waste, thereby reducing the disposal costs by two-thirds, or a savings of \$2,400,000 annually.

ARL has evaluated new water-reducible wash primers that do not contain hexavalent chromates and significantly minimize VOC- and HAP-potential emissions during coating operations. The new wash primers are water-borne acrylic latex with corrosion-inhibiting pigments. These three water-reducible acrylic latex formulations are designed for use under MIL-P-53030A (8), a water-reducible lead and chromate-free epoxy primer. The Coatings Technology Team prepared a 1200-panel matrix to evaluate these three formulations and two direct-to-metal (DTM) applications against the control specification.

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## 2. Experimental

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In this project, three vendor formulations were evaluated against the control DOD-P-15328D. The initial effort consisted of evaluating various coating candidates in a laboratory environment and selecting a suitable candidate for field testing at a renovation facility. The selected coating candidate will be applied at a renovation facility to ensure the technical practicability. The control wash primer DOD-P-15328D and the three potential replacement products were labeled, as shown in table 1.

Table 1. Control and replacement products.

D	DOD-P-15328D (control)
A	Aqua Zen by Hentzen
S	Kem Aqua by Sherwin Williams
R	RWE1033 by Spraylat
M	No pretreatment, modified primer by Niles (DTM)
P	No pretreatment, regular primer by Niles (DTM)



As shown in table 2, all substrates were coated with the water-reducible epoxy primer MIL-P-53030A. The panels exposed in Florida and Arizona were topcoated with the green CARC MIL-C-46168 (9) Type IV topcoat CARC coatings. Two sets (M and P) labeled DTM were coated just with the epoxy primer and epoxy primer plus topcoat.

Table 2. Wash primer coating systems.

Substrate	Pretreatment	Primer	Topcoat
Cold rolled steel (CRS)1080	DOD-P-15328D or one of three vendors	MIL-P-53030A epoxy primer	MIL-C-46168 (2K polyurethane topcoat)
Aluminum (Al) 2024-T3	DOD-P-15328D or one of three vendors	MIL-P-53030A	MIL-C-46168D
Al 5083-H231	DOD-P-15328D or one of three vendors	MIL-P-53030A	MIL-C-4616D
Al 6061-T3	DOD-P-15328D or one of three vendors	MIL-P-53030A	MIL-C-46168D
Al 7075-T6	DOD-P-15328D or one of three vendors	MIL-P-53030A	MIL-C-46168D

DOD-P-15328D and the three potential replacement products were applied to five different surfaces, as shown in table 3—CRS Type R 1080 panels  $4 \times 6 \times 0.032$  in and aluminum panels of alloys 2024-T3, 5083-H231, 6061-T3, and 7075-T6, at the recommended film thickness. Prior to pretreatment and testing, the panels were labeled mechanically to permanently affix the proper designation.

Table 3. Substrate used for testing.

1	CRS 1080	$4 \times 6 \times 0.032$ in
2	Al 2024-T3	$4 \times 6 \times 0.032$ in
5	Al 5083-H231	$4 \times 6 \times 0.25$ in
6	Al 6061-T3	$4 \times 6 \times 0.063$ in
7	Al 7075-T6	$4 \times 6 \times 0.032$ in

Before painting, all panels were cleaned, and the pretreatments were applied per specification. The test specimens were horizontally oriented during paint application. A conventional air-atomizing spray gun was used to apply the candidate wash primer to the appropriate substrates. Because the three vendor samples were emulsion-type systems, quite different from the control wash primer, two variations in the application of the coatings were performed. The first variation was to coat the wash primers or the epoxy primer after 1 hr and after 24 hr, a scenario that could easily occur at the depots (table 4). The second variation was to apply the recommended film thickness of the wash primer and to also apply a heavier coat of the wash primer (table 5). The full coating systems were allowed to cure at ambient temperature ( $\sim 75$  °F) and humidity for 7 days. Table 6 is a schematic detailing the test matrix.


Table 4. Application/recoat schedule.

1	Normal thickness	1 hr before primer application
2	Normal thickness	24 hr before primer application
3	Heavy thickness	1 hr before primer application
4	Heavy thickness	24 hr before primer application

Table 5. Dry film thickness.

D	DOD-P-15328D	0.5–1.0 mil
A	Aqua Zen by Hentzen Paints	0.3–0.5 mil
S	Kem Aqua by Sherwin Williams Company	0.3–0.5 mil
R	RWE-1033 by Spraylat Corporation	0.2–0.4 mil

Table 6. Test matrix.

Alloys	Pretreatments	Application	Test Method	Coupons
1080 Steel	DoD-P-15328	Thin @ 1 Hour	Outdoor Exposure (FL)	
2024 Al	Kem Aqua	Thin @ 24 Hours	Outdoor Exposure (AZ)	
5083 Al	Aqua Zen	Thick @ 1 Hour	ASTM B 117	
6061 Al	Spraylat RWE 1033	Thick @ 24 Hours	GM 9540P	
7075 Al	Direct to Metal (DTM)		ASTM D 3359	

### 3. Results and Discussion

The newly developed wash primer replacements have been extensively tested for accelerated corrosion and adhesion and have completed 2 years of outdoor exposure testing. Thus far, results are very specific to the substrate in terms of few blisters and minor fading. ARL will conduct tests on actual equipment to validate the lab and controlled testing previously completed. Current efforts are underway to field-evaluate these alternatives on actual equipment to validate the application and durability of these products. Successful completion of this test effort will lead to a revision of DOD-P-15328D, providing a qualified products list (QPL) for the new product(s). Additionally, revision of MIL-P-53030A to match the performance characteristics of new products currently in development will correspond with this initiative. Once DOD-P-15328D has been revised, the incorporation of the new wash primer products will be seamless for systems using the CARC coating.

### **3.1 Spraying Properties**

All three potential replacement products sprayed uniform films without any surface defects. The laboratory addressed two separate failure criteria by varying film thickness and application between primers. Results showed dependence on many factors including dry film thickness where heavy films slowed the curing schedule, and softer films affected intercoat adhesion.

### **3.2 Water Immersion Resistance**

American Society for Testing and Materials (ASTM) D 1308-02 (10) involves exposing an organic coating to a reagent to determine adverse affects. Coated panels were immersed in deionized water, half of the panel length, at room temperature ( $23 \pm 5$  °C) for 7 days. Immediately upon removal, and after a 24-hr recovery period, the panels were examined for any defects, such as blistering, loss of adhesion, color, and gloss change. All panels passed the water immersion test.

### **3.3 Flexibility**

The Mandrel Bend Test was performed on all coatings in accordance with ASTM D 522-93 (11). The purpose of this test was to rate each coating's resistance to cracking and rate the flexibility of each coating. This test demonstrated that all coatings were generally flexible, but most showed very minor cracks. Due to panel thickness, Al 6061 and 7075 alloys were not tested.

### **3.4 Impact Resistance**

The standard test for resistance to deformation (impact) was performed using a Gardner height impact tester, consisting of a vertical tube to guide a cylindrical weight that is dropped on a punch resting on the test panel. Impact resistance can be described as a paint property that quantitatively characterizes the durability of a coating with respect to a rapid impact event. After curing 7 days at ambient laboratory conditions, the impact-resistance test based on ASTM D 2794 (12) was performed on all coatings using 40 in-lb weights. All selected coatings passed using the 40 in-lb weights, with minor distinctions when impact was increased to 80 in-lb. Results are listed in table 7.

### **3.5 Dry Adhesion Testing (ASTM D 3359-93 Method B Adhesion Testing) (13)**

Table 8 lists the panels used in adhesion testing and the results. The ASTM cross-cut adhesion testing was performed with 2-mm line spacing, appropriate for dry film thickness between 2 and 5 mil (1 mil = 0.001 in).

### **3.6 Wet Adhesion Testing**

All samples were immersed in water for 7 days and subjected to cross-cut adhesion testing. Upon removal and after a 24-hr recovery period, samples were evaluated for blistering, softening, and loss of adhesion. Table 9 lists the panels used in adhesion testing and their results.

Table 7. Results of impact resistance.

		<b>GM 9540 100 Cycles + 360 Days Ambient</b>
<b>Panel</b>	<b>360 Days Ambient Reverse Impact (80 in-lb)</b>	<b>Reverse Impact (80 in-lb)</b>
1M	2	2
1P	2	2
1D	2	1
1R	5	5
1A	5	5
1S	2	3
6M	1	—
6P	1	—
6D	2	—
6R	5	—
6A	5	—
6S	2	—
7M	2	—
7P	2	—
7D	3	—
7R	5	—
7A	5	—
7S	3	—
	<b>Reverse Impact (40 in-lb)</b>	<b>Reverse Impact (40 in-lb)</b>
2M	2	4
2P	1	4
2D	*3	*3
2R	5	5
2A	5	4
2S	4	3

Rating Criteria: 1 = 80%–90% removal of coating/large popping.

2 = 70%–80% removal of coating.

3 = 60%–70% removal of coating.

4 = 10% removal/cracking.

5 = <5% removal of coating.

The ASTM cross-cut adhesion testing was performed with 2-mm line spacing, appropriate for dry film thickness between 2 and 5 mil (1 mil = 0.001 in). All samples passed the dry adhesion test.

### 3.7 Accelerated Corrosion Resistance

Accelerated corrosion testing was performed using both a neutral salt spray test per ASTM B 117 (14) and an accelerated cyclic corrosion test per General Motors (GM) 9540P (15). Salt spray resistance was widely used by the paint industry as a quality control test and was not necessarily indicative of long-term performance of a coating. Exposure to salt fog provided information about diffusion and porosity of the coating. Prior to exposure, the panels for each system were all scribed with two intersecting scribes (“X”) through the coatings to the substrate.

Table 8. Dry adhesion testing.

Pretreatment	CRS 1080	Al 2024-T3	Al 5083-H231	Al 6061-T3	Al 7075-T6
Aqua Zen	5B (pass)	5B (pass)	5B (pass)	5B (pass)	5B (pass)
DOD-P-15328D	5B (pass)	5B (pass)	5B (pass)	5B (pass)	5B (pass)
Kem Aqua (K)	4B (pass)	5B (pass)	5B (pass)	5B (pass)	5B (pass)
RWE-1033	5B (pass)	5B (pass)	5B (pass)	5B (pass)	5B (pass)
Kem Aqua (S)	5B (pass)	4B (pass)	5B (pass)	5B (pass)	5B (pass)
MIL-P-53022-10 w/additive	2B(fail)	1B (fail)	1B (fail)	4B (pass)	3B (fail)
MIL-P-53022-10 Direct to metal	5B (pass)	2B (fail)	1B (fail)	3B (fail)	2B (fail)

Notes: 5B rating means no removal.

4B rating means <5% removal.

3B rating means 5%–15% removal.

2B rating means 15%–35% removal.

1B rating means 35%–65% removal.

Table 9. Wet adhesion testing.

Pretreatment	CRS 1080	Al 2024-T3	Al 5083-H231	Al 6061-T3	Al 7075-T6
Aqua Zen	5B (pass)	5B (pass)	5B (pass)	5B (pass)	5B (pass)
DOD-P-15328D	5B (pass)	5B (pass)	5B (pass)	5B (pass)	5B (pass)
Kem Aqua (K)	4B (pass)	5B (pass)	5B (pass)	5B (pass)	5B (pass)
RWE-1033	5B (pass)	5B (pass)	5B (pass)	5B (pass)	5B (pass)
Kem Aqua (S)	5B (pass)	4B (pass)	5B (pass)	5B (pass)	5B (pass)
MIL-P-53022-10 w/additive	2B(fail)	2B (fail)	1B (fail)	1B (fail)	4B (pass)
MIL-P-53022-10 Direct to metal	5B (pass)	1B (fail)	1B (fail)	1B (fail)	1B (fail)

The panels were X-scribed using a standard carbide-tipped hardened steel scribe. The painted panels (three each) for each coating were exposed to ASTM B 117 neutral salt fog conditions consisting of 95 °F with saturated humidity and atomized fog of 5% sodium chloride solution. The test panels were rated for damage at weekly intervals and inspected at 500-hr intervals up to 2088 hr of salt spray. All the painted panels appeared visually identical before testing. Panels were evaluated using ASTM D 1654 (16) for “Standard Test Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments” and ASTM D 714-87 (17) for “Standard Test Method for Evaluating Degree of Blistering of Paints.” Final detailed ratings for the 2088-hr duration using ASTM D 1654 quantitatively indicated the damage caused by pitting or delamination outward from the scribe.

GM 9540P is an accelerated cyclic corrosion test that was developed by the automotive industry to replicate long-term outdoor performance of coatings more accurately than the conventional salt spray test. It evaluates adhesion as well as corrosion of the system over time. A cyclic corrosion test chamber (CCTC) was used to perform the GM 9540P test. The test consisted of the repetition of one cycle with 18 separate stages including salt (1.25% by mass: 0.9% NaCl, 0.1% CaCl<sub>2</sub>, and 0.25% NaHCO<sub>3</sub>) water mist, humidity, drying, ambient, and heated drying.

This process repeated 80 times to a scribed panel is claimed by industry to be equivalent to 10 years of field exposure in South Florida for their specific coating systems. For this test, the groups of scribed coupons were exposed until failure or completion of 100 cycles. In addition, standard plain carbon steel calibration coupons described in GM 9540P and supplied by GM were initially weighed and subsequently monitored for mass loss at intervals set by the specification. Mass losses measured for steel coupons used for this test were within parameters stated in the GM 9540P specification. For each coating tested, three panels were subjected to CCTC testing. As in salt spray, the panels were X-scribed. The scribed panels were placed into the chamber and tested using GM 9540P, Method B10, which provided a more realistic accelerated environmental test than conventional salt spray.

The criteria for failure was either creep from scribe of  $>10$  mm (ASTM D 1654 rating of  $<3$ ) or an ASTM D 714-87 rating for blistering in excess of GM 9540P in the unscribed regions. Upon removal, coupons were rinsed in deionized water, and scraped at a  $30^\circ$  contact angle to the test surface with a blunt edge, metal knife prior to evaluation. Once scraped, the test panels were removed from other corrosion testing.

Al 2024-T3 is mainly used on aircraft and missiles and is the least corrosive resistant of the series. For Al 2024, as shown in table 10, the alternative systems have 40%–60% of the control protection after 1088 hr in salt fog, while DTM has 10%, and at 2088 hr, DTM has 0% protection. At 44 cycles in the GM 9540P, the control offers 90% protection, while the three alternative systems offer ~50%, and the DTM offers ~10%. The separation between 44 cycles and 100 cycles was marginal.

Al 5083-H231 does well in corrosion due to its protective oxide layer and is normally utilized on armor and tactical ground systems. In table 11, the slight change in data from 44 to 100 cycles in the GM chamber shows that two vendors are equal to the control. In ASTM B 117, the three vendors and DTM offer 30% protection of the control after 2088 hr.

Al 6061-T3 is mainly used on components, and as seen in table 12, all the pretreatments on Al 6061 performed well with little or no damage to the scribed region. DTM has 0% protection at 44 and 100 cycles, while the three vendors and the control offer 90% of corrosion protection. Additionally, the salt fog environment demonstrates that the three vendors and the control provide 90% protection, while the DTM offers 50%.

Most pretreatments on Al 7075-T6 performed well, with little or no damage to the scribed region. The top performers are clearly visible in table 13. Except for the DTM, all pretreatment surfaces had very minute creepage or corrosion.

The coated steel panels performed differently from the aluminum alloys, with greater rust and surface corrosion. The distinction is shown in table 14 for 1000 hr in salt fog, where the three vendors and the DTM are inferior to the control. At 2088 hr, the data confirm that the wash primer will extend life extension and protection.

Table 10. Average creepage from scribe (millimeter) for Al 2024-T3.

ASTM B 117			GM 9540P		
Al 2024-T3	1088 hr	2088 hr	Al 2024-T3	44 Cycles	100 Cycles
2D1	10.00	9.33	2D1	9.33	8.00
2D2	9.33	8.00	2D2	7.00	5.00
2D3	9.33	9.33	2D3	7.66	6.00
2D4	9.00	9.00	2D4	6.66	5.67
—	9.42	8.92	—	7.66	6.17
2A1	5.67	0.00	2A1	7.67	4.33
2A2	6.00	4.33	2A2	6.66	3.67
2A3	5.00	4.67	2A3	7.00	4.33
2A4	6.00	3.67	2A4	5.66	3.00
—	5.67	3.00	—	6.75	3.83
2S1	4.00	0.33	2S1	5.33	4.67
2S2	6.00	1.00	2S2	5.33	3.33
2S3	6.00	1.00	2S3	6.00	4.67
2S4	6.00	1.00	2S4	6.00	4.67
—	4.15	3.58	—	5.67	4.34
2R1	3.33	9.33	2R1	6.00	6.00
2R2	2.67	9.00	2R2	5.66	5.00
2R3	4.67	10.00	2R3	5.66	5.00
2R4	4.00	7.67	2R4	5.00	4.00
—	3.67	3.30	—	5.58	5.00
2M2	1.00	0.00	2M2	1.00	0.00
2M4	0.00	0.00	2M4	2.67	0.00
—	0.00	0.00	—	1.84	0.00
2P2	1.67	0.00	2P2	3.00	2.66
2P4	0.60	0.00	2P4	3.00	2.66
—	1.14	0.00	—	3.00	2.66

For the GM 9540P specification, 100 cycles was the best separator, with the DTM allowing 0% protection, and the better performers on steel were easily observed. Most films were intact outside the scribed areas. The four treated surfaces showed similar corrosion degradation at the scribed areas and no evidence of blisters. The untreated surface (DTM) showed 90% delamination of the panel. Figures 1–6 show examples of samples exposed in both chambers.

### 3.8 Outdoor Weathering in Florida and Arizona Exposure

The outdoor exposure testing was performed at the Miami, FL site (26° N), at a tilt of 5° from the horizontal facing south. The samples were mounted on an aluminum rack, with the coated side facing the sun. The total radiant energy was measured at 12,384.16 MJ/m<sup>2</sup>, 295,988 langley, and the ultraviolet (UV) measured at 525.23 MJ/m<sup>2</sup>. Table 15 summarizes results after 24 months of exposure, with visual color change rated at 4f for all, where 4 = pronounced and f = fade. Corrosion and blisters were noted on the pretreated steel substrate and graded 6f, where 6 = blister size of 1 mm and f = few. Except for sample A that had 10 of 12 panels and sample S

Table 11. Average creepage from scribe (millimeter) for Al 5083-H231.

ASTM B 117			GM 9540P		
Al 5083-H231	1088 hr	2088 hr	Al 5083-H231	44 Cycles	100 Cycles
5D1	9.33	9.00	5D1	10.00	10.00
5D2	9.00	9.00	5D2	9.67	9.33
5D3	9.00	9.00	5D3	9.67	9.67
5D4	9.00	9.00	5D4	9.33	9.00
—	9.08	9.00	—	9.67	9.50
5A1	7.00	6.60	5A1	9.00	9.00
5A2	7.67	7.62	5A2	9.00	9.00
5A3	9.00	9.00	5A3	9.00	9.00
5A4	9.00	9.00	5A4	9.00	9.00
—	8.17	3.00	—	9.00	9.00
5S1	9.00	9.00	5S1	8.33	8.00
5S2	7.67	7.00	5S2	8.00	7.67
5S3	9.00	9.00	5S3	7.00	7.00
5S4	7.67	7.33	5S4	9.00	9.00
—	4.15	3.58	—	8.08	7.92
5R1	9.67	9.33	5R1	9.33	9.00
5R2	9.33	9.33	5R2	9.00	7.67
5R3	10.00	10.00	5R3	9.00	8.67
5R4	9.33	9.00	5R4	9.33	8.67
—	9.58	3.30	—	9.17	8.50
5M2	3.33	2.67	5M2	6.33	5.33
5M4	5.00	4.33	5M4	5.33	0.00
—	0.00	0.00	—	5.83	0.00
5P2	3.33	2.00	5P2	5.33	5.33
5P4	5.33	4.67	5P4	6.00	5.67
—	4.33	3.34	—	5.67	5.50

with 11 of 12 panels showing blisters, all other samples showed no defects. Samples 1M, 1P, 1R, and 1S had severe creep from the scribe. Figures 7 and 8 represent samples that were exposed in Florida.

Exposure testing was also performed in New River, AZ (34° N) at a tilt angle of 5° from the horizontal facing south. The samples were mounted unbacked on a continuous exposure rack, with the coated side facing the sun. The total radiant energy measured at 18,057.96 MJ/m<sup>2</sup>, 431,595 langley, and the UV measured at 770.36 MJ/m<sup>2</sup>. Various panels showed some flaking at the center of the X score. All the substrates had pronounced (4) discoloration (DC) and fading (F), and at 24 months, DTM (2M\*, 2P\*) exhibited slight delamination at the scribe with no run down (table 16). Approximately 280 MJ/m<sup>2</sup> of UV is equivalent to 1 year of outdoor exposure in southern Florida.



Table 12. Average creepage from scribe (millimeter) of Al 6061-T3.

ASTM B 117			GM 9540P		
Al 6061-T3	1080 hr	2088 hr	Al 6061-T3	44 Cycles	100 Cycles
6D1B	9.00	9.00	6D1W	10.00	9.00
6D2B	9.00	9.00	6D2W	10.00	9.00
6D3B	9.00	10.00	6D3W	10.00	9.00
6D4B	9.00	10.00	6D4W	9.00	9.00
—	9.20	9.60	—	9.80	9.20
6A1B	7.00	9.00	6A1W	10.00	9.00
6A2B	9.00	9.00	6A2W	10.00	6.00
6A3B	8.00	8.00	6A3W	10.00	10.00
6A4B	9.00	8.00	6A4W	10.00	9.00
—	8.25	8.50	—	10.00	8.50
6R1B	10.00	10.00	6R1W	10.00	4.00
6R2B	10.00	10.00	6R2W	10.00	8.00
6R3B	9.00	9.00	6R3W	10.00	9.00
6R4B	9.00	9.00	6R4W	10.00	9.00
—	9.50	9.50	—	10.00	7.50
6S1B	8.00	4.00	6S1W	9.00	4.00
6S2B	9.00	9.00	6S2W	9.00	0.00
6S3B	9.00	6.00	6S3W	9.00	3.00
6S4B	9.00	6.00	6S4W	9.00	3.00
—	8.75	6.25	—	9.00	2.50
6M2B	4.00	0.00	6M2W	3.00	0.00
5M4B	3.00	0.00	5M4W	6.00	0.00
—	3.50	0.00	—	4.50	0.00
6P2B	6.00	0.00	6P2W	3.00	0.00
6P4B	6.00	0.00	6P4W	4.00	4.00
—	6.00	0.00	—	3.50	2.00

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## 4. Summary and Conclusion

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Laboratory results and a wide range of data exist for the four different systems and five substrates. The results show dependence on many factors including substrate/material coating thickness, condition of testing, and the interpretation of results. Pretreatment performances vary among alloys. So far, the replacements are similar in performance to the control DOD-P-15328D wash primer, and the results are very promising. Of course, no single laboratory test can provide all the answers. The result of replacing the DOD material with one of the alternative systems is imminent, and the impact of this change will be positive across the board.

Affected installations, facilities, and weapons systems will include all tactical combat vehicles and U.S. Army aviation helicopters and equipment, and depots that are currently looking for viable alternative hexavalent chromate free wash primers.

Table 13. Average creepage from scribe (millimeter) of Al 7075-T6.

ASTM B 117			GM 9540P		
Al 7075-T6	1080 hr	2088 hr	Al 7075-T6	44 Cycles	100 Cycles
7D1B	10.00	9.00	7D1B	10.00	9.00
7D2B	9.00	9.00	7D2B	10.00	9.00
7D2B	9.00	9.00	7D2B	9.00	9.00
7D3B	10.00	10.00	7D3B	10.00	10.00
7D4B	9.00	9.00	7D4B	9.00	9.00
—	9.40	9.20	—	9.60	9.20
7A1B	10.00	9.00	7A1B	10.00	9.00
7A2B	9.00	9.00	7A2B	10.00	9.00
7A3B	10.00	10.00	7A3B	10.00	10.00
7A4B	9.00	9.00	7A4B	9.00	9.00
—	9.50	9.25	—	9.75	9.25
7R1B	9.00	9.00	7R1B	10.00	9.00
7R2B	9.00	9.00	7R2B	10.00	9.00
7R3B	10.00	10.00	7R3B	10.00	10.00
7R4B	9.00	9.00	7R4B	9.00	9.00
—	9.25	9.25	—	9.75	9.25
7S1B	9.00	9.00	7S1B	9.00	9.00
7S2B	7.00	6.00	7S2B	7.00	6.00
7S3B	9.00	9.00	7S3B	9.00	9.00
7S4B	7.00	6.00	7S4B	7.00	6.00
—	8.00	7.50	—	8.00	7.50
7M2B	3.00	2.00	7M2B	3.00	2.00
7M4B	4.00	3.00	7M4B	6.00	3.00
—	3.50	2.50	—	4.00	2.50
7P2B	2.00	2.00	7P2B	3.00	2.00
7P4B	7.00	6.0.0	7P4B	7.00	5.00
—	4.50	3.50	—	5.00	3.50

The elimination of hexavalent chromium and much of the solvent in wash primer would have a direct positive impact on worker health and safety. It will eliminate 12,600 lb of zinc chromate, 16,800 gal of package solvents, and 18,900 gal of thinner solvents emitted annually as the result of DOD-P-15328D. In addition, it will help to eliminate the need to dispose of 6,000,000 lb of CARC-stripped wastes as hazardous wastes. At this point, the major challenge is to reconcile the differences observed between accelerated weathering and the natural world, because there has been no coating failure after 2 years at the two exposure sites. The natural environmental testing will continue for another year and will be compared to the simulated, controlled laboratory results.

Table 14. Average creepage from scribe (millimeter) for CRS 1080.

ASTM B 117			GM 9540P		
CRS 1080	1080 hr	2088 hr	CRS 1080	44 Cycles	100 Cycles
1D1	3.33	2.33	1D1	—	—
1D2	4.67	3.33	1D2	4.67	3.00
1D3	4.33	2.33	1D3	5.00	3.00
1D4	5.00	4.00	1D4	5.00	4.33
—	4.33	3.00	—	4.89	3.44
1A1	3.00	0.00	1A1	4.33	1.67
1A2	2.00	0.00	1A2	4.33	1.67
1A3	1.33	0.67	1A3	4.67	2.00
1A4	5.33	2.00	1A4	4.33	2.67
—	2.92	2.10	—	4.42	2.50
1S1	3.33	3.33	1S1	2.67	0.00
1S2	4.33	4.00	1S2	3.33	0.00
1S3	4.67	3.33	1S3	3.00	1.00
1S4	4.30	3.67	1S4	3.00	0.33
—	4.15	3.58	—	3.00	1.00
1R1	3.33	1.00	1R1	5.00	4.00
1R2	2.67	2.00	1R2	5.00	3.67
1R3	4.67	3.33	1R3	5.00	3.00
1R4	4.00	2.33	1R4	4.33	3.33
—	3.67	3.30	—	4.83	3.50
1M2	0.00	0.00	1M2	1.00	0.00
1M4	0.00	0.00	1M4	2.67	0.00
—	0.00	0.00	—	1.84	0.00
1P2	1.67	0.00	1P2	3.67	0.00
1P4	3.00	0.67	1P4	3.67	0.33
—	2.34	0.34	—	3.67	0.17

Table 15. South Florida exposure at 24 months.

Specimen ID	Color Change	Face Rust Wash	Scribe Rust Unwashed	Crack Unwashed	Blisters Unwashed	Delamination Unwashed
1A	4f	10	10	10	(10/12)	10
1D	4f	10	10	10	(3/12)	10
1M	4f	10	2:1/16	10	10	10
1P	4f	10	2:1/16	10	10	10
1R	4f	10	2	10	(2/12)	10
1S	4f	10	2	10	(11/12)	10
2A	4f	10	10	10	10	10
2D	4f	10	10	10	10	10
2K	4f	10	10	10	10	10
2M	4f	10	10	10	10	10
2P	4f	10	10	10	10	6
2R	4f	10	10	10	10	10
2S	4f	10	10	10	10	10
5A	4f	10	10	10	10	10
5D	4f	10	10	10	10	10
5K	4f	10	10	10	10	10
5M	4f	10	10	10	10	10
5P	4f	10	10	10	10	10
5R	4f	10	10	10	10	10
5S	4f	10	10	10	10	10

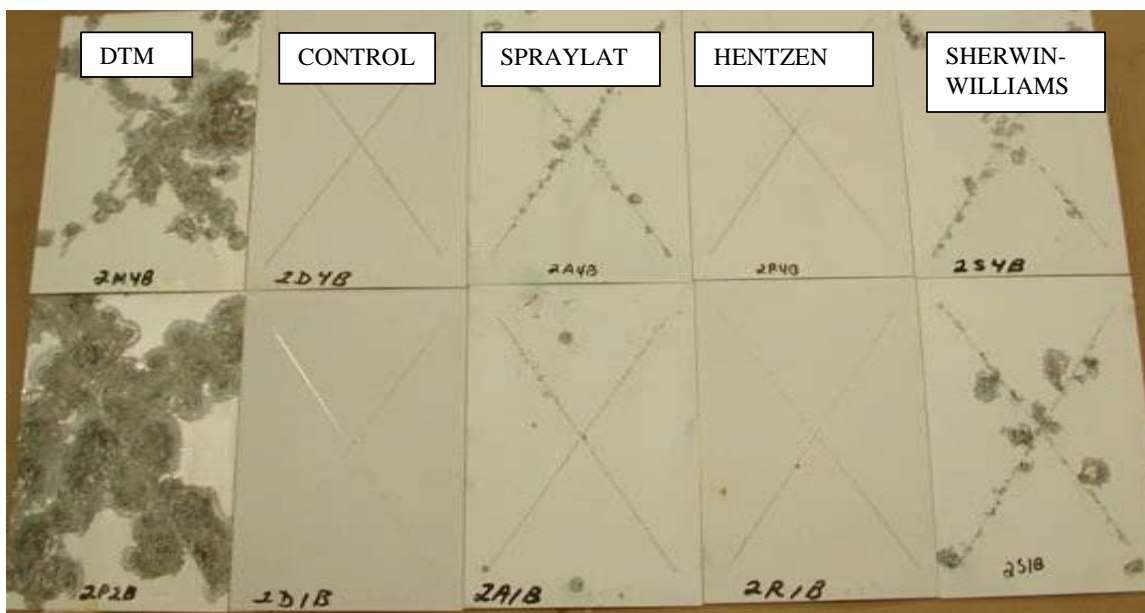


Figure 1. GM 9540P results on Al 2024-T3.

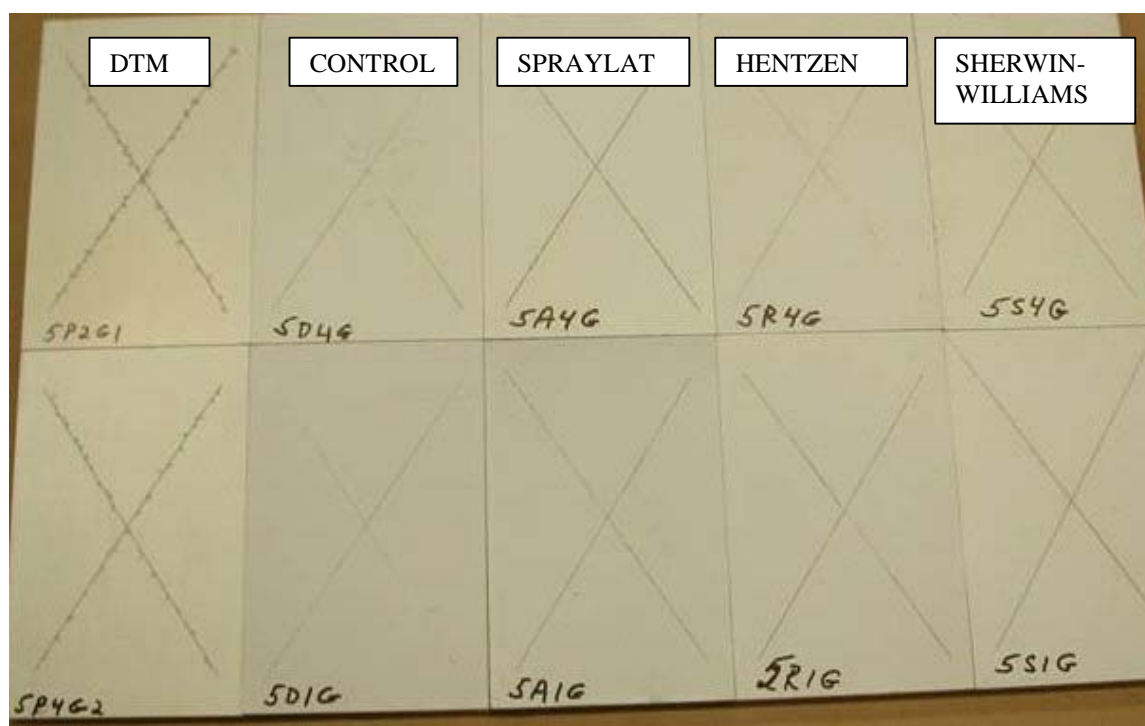


Figure 2. GM 9540P results for Al 5083-H231.

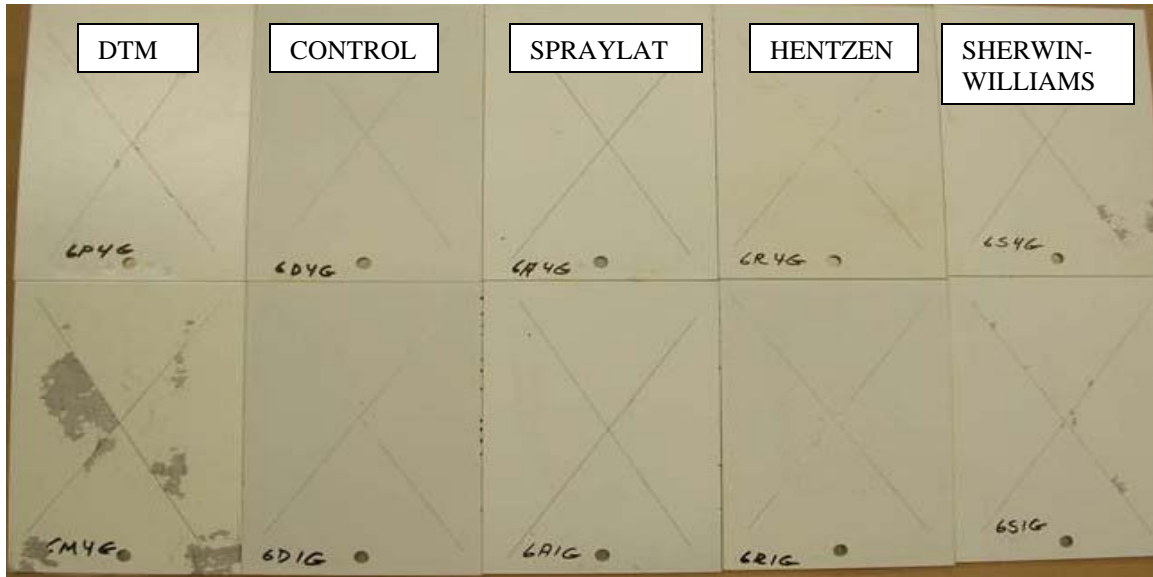


Figure 3. GM 9540P results on Al 6061-T3.

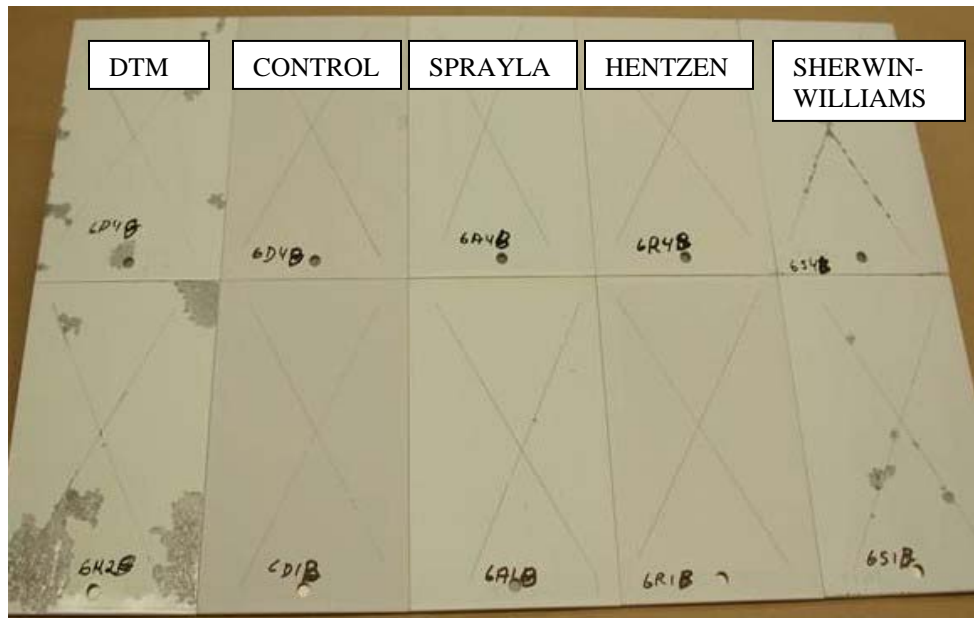


Figure 4. ASTM B 117 results on Al 6061-T3.



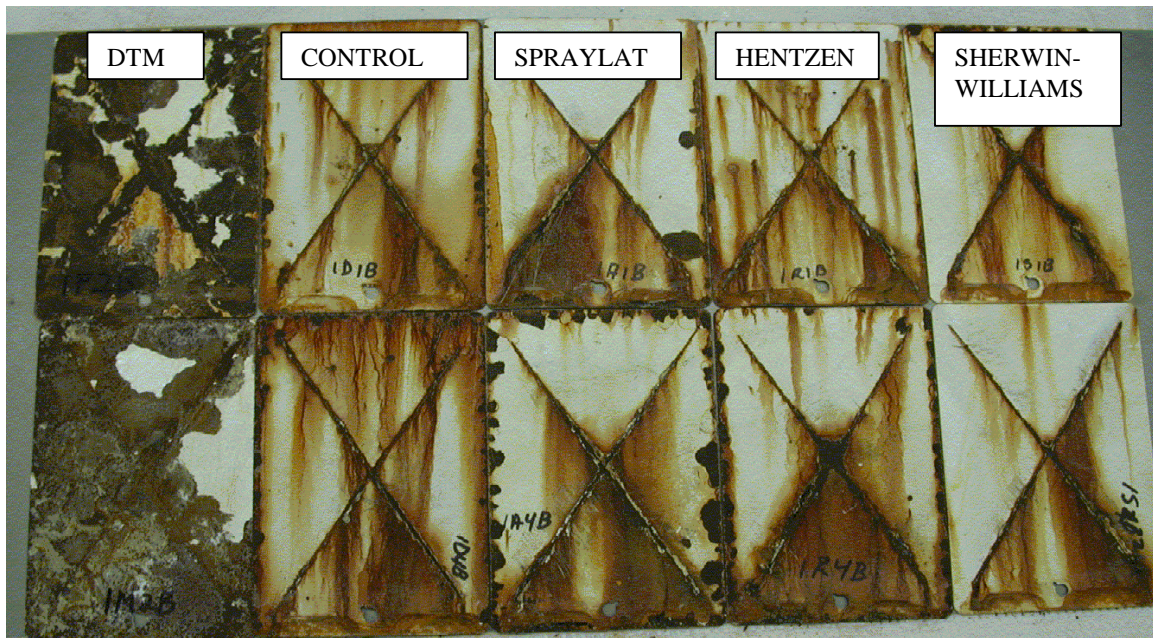


Figure 5. ASTM B 117 results on CRS 1080.

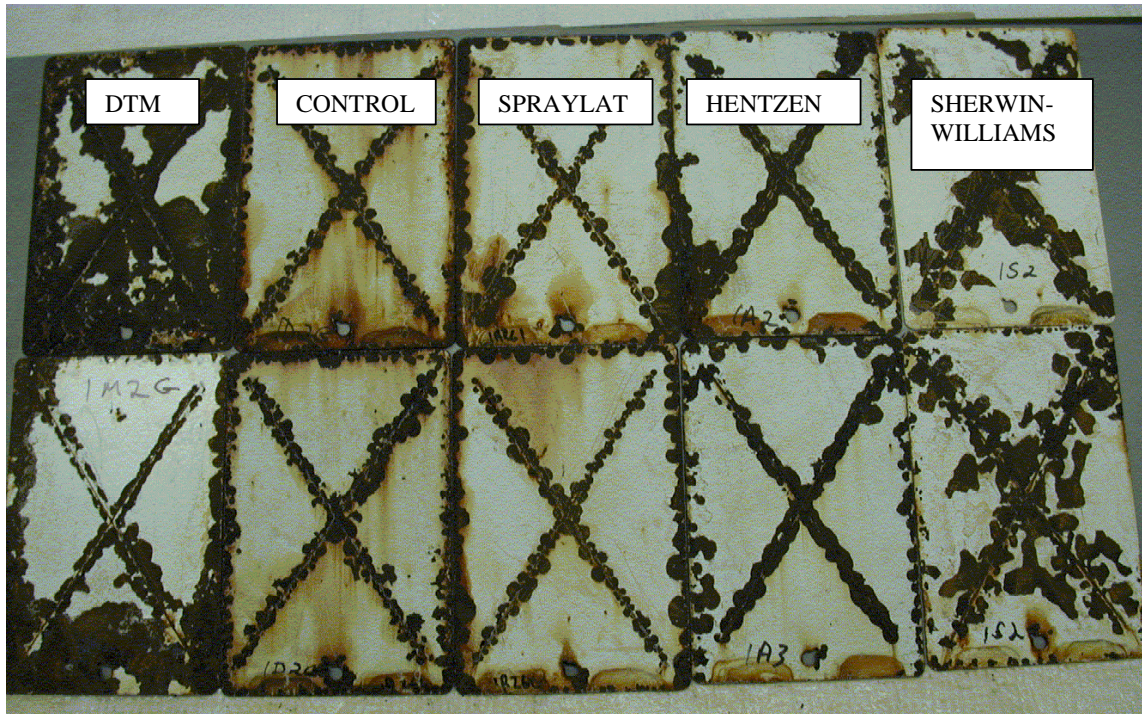


Figure 6. GM 9540P results on CRS 1080.



Figure 7. Vendor 613 days 1S2 F2.



Figure 8. Control after 613 days 1D4 F1.

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## 5. References

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1. Union Carbide Corporation. Bakelite Vinyl Butyral Resins. *Union Carbide Coatings Materials* **1971**.
2. DOD-P-15328D. *Primer (Wash), Pretreatment (Formula No. 117 for Metals)* **2001**.
3. Angelmayer, K. H. Chromate-Free, Wash Primers Problems and Possible Solutions. *Polymers Paints and Colour Journal* **1986**.
4. MIL-P-53072C. *Chemical Agent Resistant Coating, (CARC) System Application Procedures and Quality Control Inspection* **1992**.
5. TT-C-490. *Chemical Conversion Coatings and Pretreatments for Ferrous Surfaces* **2002**.
6. Whiting, L. R. *Corrosion*, June 1989.
7. Lombourne R., Ed. Paint and Surface Coatings. *Applied Science and Industrial Technology*; University of Bristol: West Sussex, England, 1987.
8. MIL-P-53030A. *Primer, Epoxy Coating, Water Reducible, Lead and Chromate Free* **1992**.
9. MIL-C-46168. *Coatings, Aliphatic Polyurethane Chemical Agent Resistance Coatings (CARC)* **1993**.
10. ASTM D 1308-02. Standard Test Method for Effect of Household Chemicals on Clear and Pigmented Organic Finishes. *Annu. Book ASTM Stand.* **2002**.
11. ASTM D 522-93. Standard Test Methods for Mandrel Bend Test of Attached Organic, Coatings. *Annu. Book ASTM Stand.* **1993**.
12. ASTM D 2794. Standard Test Method for Resistance of Organic Coatings to the Effects of Rapid Deformation (Impact). *Annu. Book ASTM Stand.* **1993**.
13. ASTM D 3359-93. Standard Test Method for Measuring Adhesion by Tape Test. *Annu. Book ASTM Stand.* **1998**.
14. ASTM B 117. Standard Method of Salt Spray (Spray) Testing. *Annu. Book ASTM Stand.* **1990**.
15. GM 9540P. *Accelerated Corrosion Test*, General Motors Engineering Standards **1997**.
16. ASTM D 1654. Standard Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments. *Annu. Book ASTM Stand.* **1984**.
17. ASTM D 714-87. Standard Test Method for Evaluating Degree of Blistering of Paints. *Annu. Book ASTM Stand.* **1987**.



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